2006 ERSD Annual Report
DOE-BER Environmental Remediation Sciences Project # 94986

Ion Recognition Approach to Volume Reduction of Alkaline Tank Waste by Separation of Sodium Salts

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Summary

The purpose of this research involving collaboration between Oak Ridge National Laboratory (ORNL) and Pacific Northwest National Laboratory (PNNL) is to explore new approaches to the separation of sodium hydroxide, sodium nitrate, and other sodium salts from high-level alkaline tank waste. The principal potential benefit is a major reduction in disposed waste volume, obviating the building of expensive new waste tanks and reducing the costs of low-activity waste immobilization. Principles of ion recognition are being researched toward discovery of liquid-liquid extraction systems that selectively separate sodium hydroxide and sodium nitrate from other waste components. The successful concept of pseudohydroxide extraction using fluorinated alcohols and phenols is being developed at ORNL and PNNL toward a greater understanding of the controlling equilibria, role of solvation, and of synergistic effects involving crown ethers. Studies at PNNL are directed toward new solvent formulation for the practical sodium pseudohydroxide extraction systems.

Research Objectives

The primary purpose of this project is to gain new fundamental knowledge that can ultimately point to practical approaches to the reduction of the volume of radioactive tank waste that must be immobilized for disposal, especially in large centralized processing plants such as the Hanford Waste Treatment Plant. The overall goal is to provide the scientific foundation upon which the feasibility of liquid-liquid extraction chemistry can be evaluated for separation of sodium hydroxide, sodium nitrate, and other sodium salts (e.g., nitrite and sulfate) from alkaline tank waste. Toward this end, a collaboration between ORNL (B. A. Moyer and P. V. Bonnesen) and PNNL (T. G. Levitskaia and G. J. Lumetta) combines expertise and special facilities in organic synthesis (ORNL), solvent extraction (ORNL and PNNL), and radioactive waste processing (PNNL). Our approach aims to gain insights at the molecular level and challenge these insights as far as simple experiments on actual waste.

This work aims for a principal potential benefit in terms of a major reduction in vitrified waste volume by removal of soluble sodium electrolytes, which constitute most of the mass of the tank-wastes. As such, sodium has a direct bearing on the amount of waste that must be immobilized for disposal, and some anions of its salts play special roles in waste treatment. For example, nitrate leaching from waste forms such as grout limit their performance and acceptability; sulfate has a low solubility in borosilicate glass melts; and hydroxide has potential value for recycle (e.g., in waste retrieval and sludge pretreatment).

In terms of the science that is needed to discover applicable liquid-liquid separation systems, basic questions that this research addresses include whether it is even possible to remove bulk

sodium electrolytes from the waste, what types of chemical systems might be applicable, what functional properties might be obtained, how those functional properties might be understood based on molecular properties, what selectivity might be possible, and how the extraction could be reversed to regenerate the solvent and to recover a usable sodium product stream. In the previous and current funding periods, all of these questions have been in part successfully addressed, with the emergence of some promising systems and associated understanding. Nevertheless, understanding is incomplete, and the overall problem of sodium removal is not resolved.

The overall problem is complex in that multiple sodium electrolytes are involved, and generality has not yet been achieved. For example, sodium hydroxide may now be selectively separated from the waste based on our results, an important and useful milestone in itself, but the extraction of the other bulk sodium electrolytes in the waste, such as sodium nitrate, nitrite, and sulfate, has not yet been satisfactorily explored and remains impractically weak. In addition, there are outstanding issues regarding sodium hydroxide extraction that must be addressed for closure in this area, including a successful proof-of-principle demonstration in a simple real-waste test for facilitating technology transfer to potential users.

This project addresses the OBER long-term environmental-remediation goal "Develop science-based solutions for cleanup and long-term monitoring of Department of Energy (DOE) contaminated sites." It is clearly aimed toward development of a science-based solution to tank-waste cleanup, with potentially large cost savings.

Research Progress

This annual report covers progress achieved at PNNL in FY 2006. A separate report will be issued reflecting progress to date for work at ORNL. The work conducted at PNNL in FY 2006 continued to focus on developing a pseudohydroxide extraction (PHE) system that utilizes a process-friendly diluent. Pseudohydroxide extraction employs a weak lipophilic organic acid (e.g., a phenol or fluorinated alcohol; represented in this discussion as HA) capable of proton exchange with the aqueous sodium ion at elevated pH. The lipophilic acid is dissolved in a water-immiscible diluent to form the process solvent and is converted to the sodium form upon contact with NaOH solution. Upon subsequent contact with water, the organic-phase sodium salt releases the Na^+ cation via hydrolysis, thereby producing an aqueous solution of NaOH and regenerating the organic acid. Spectral studies have confirmed that the mechanism of PHE involves the deprotonation of the alcohol to yield the corresponding alkoxide in the organic phase (Maya, Moyer, and Lance 2003).

Cation-exchange processes involving sodium ion are often hindered by the highly positive Gibbs energy of its partitioning into typical water-immiscible solvents (Marcus 1997), and choosing a suitable diluent becomes particularly important. The majority of recently reported investigations of NaOH extraction *via* PHE employed 1-octanol diluent, which exhibits donor-acceptor properties favorable for the stabilization of the deprotonated alkoxide and Na^+ extraction in comparison with other water-immiscible solvents (Chambliss et al. 2002, Haverlock et al. 2003). The ion-pair extraction mechanism for the NaOH partitioning was confirmed by vibrational and mass spectroscopic techniques (Kang et al. 2005). Although the donor-acceptor properties of 1-octanol are well-suited for PHE, its ability to solubilize appreciable amounts of water and its high viscosity are undesirable in an industrial application. At high sodium loading, expansion of the organic phase volume was observed with 1-octanol as the diluent (Haverlock et

al. 2003), and in some cases, gelling of the loaded solvent occurred (Lumetta et al. 2002). Aliphatic diluents are usually preferred for industrial solvent extraction processes. However, they entail trade-offs, such as poor solvation of the ionic species leading to very weak extraction, particularly of the alkali metal ions. Also the solubilities of the lipophilic alcohols in the aliphatic diluents are generally not high enough to yield practical PHE systems. In FY 2005, we investigated the possibility of using a commercially available aliphatic hydrocarbon diluent (Isopar® L) modified with 1-octanol to overcome the limitations of the previously described PHE solvent formulations.

This solvent—consisting of 1 M 3,5-di-*tert*-butylphenol (35-DTBP) plus 1 M 1-octanol dissolved in Isopar® L—combined the donor-acceptor properties of 1-octanol with the more favorable physicochemical characteristics of the aliphatic diluent. Various spectroscopic investigations and liquid-liquid equilibrium modeling indicated the sodium extraction mechanism for the 1-octanol modified PHE solvent could be described by the following reactions:

$$Na^+(aq) + OH^-(aq) \leftrightarrow NaOH(org) \qquad K_1 \qquad (1)$$

$$Na^+(aq) + OH^-(aq) \leftrightarrow Na^+(org) + OH^-(org) \qquad K_2 \qquad (2)$$

$$Na^+(aq) + OH^-(aq) + HA(org) \leftrightarrow NaA(org) + H_2O \qquad K_3 \qquad (3)$$

$$Na^+(aq) + OH^-(aq) + 2HA(org) \leftrightarrow NaA_2H(org) + H_2O \qquad K_4 \qquad (4)$$

where HA refers to the protonated form of 35-DTBP (Lumetta and Levitskaia, 2006).

In FY06, to further the pursuit for an industrially practical PHE process, we replaced the 1-octanol modifier used in the previous solvent formulation with Exxal® 8. The latter is a commercial-grade alcohol (predominantly isooctyl), suitable for large-scale industrial application. The 35-DTBP cation exchanger was used in this work. The moderately high acidity of 35-DTBP (aqueous $pK_a = 9.9$) ensures effective proton/sodium exchange at reasonable aqueous-phase NaOH concentrations and successful regeneration of the phenol by hydrolysis upon contact with water. These properties make 35-DTBP especially suitable for the PHE. Previous comparative studies have demonstrated 35-DTBP to be one of the most efficient sodium extractants in several cation-exchange based extractive systems (Chambliss et al. 2002, Levitskaia et al. 2003). In addition, 3,5-DTBP exhibited improved phase behavior in comparison with other phenols (Haverlock et al. 2003). The new solvent formulation consisted of 1 M 35-DTBP and 15 wt% Exxal® 8 in Isopar® L. This organic phase solution was used to develop and demonstrate the PHE process concept, and the results of these studies are reported here.

In the previous work the PHE solvent was formulated as 1 M 35-DTBP plus 1 M 1-octanol in Isopar® L (Lumetta and Levitskaia 2006). Because Exxal® 8 is a mixture of higher-molecular weight alcohols, formulating the solvent on a mol/L basis is not straightforward. At 1 M the 1-octanol content is ~16 wt%, so 15 wt% Exxal® 8 was chosen for the new PHE solvent formulation. This was expected to give comparable performance. The extraction behavior of 35-DTBP in Isopar® L modified with 15 wt% Exxal® 8 and 1 M 1-octanol was compared by systematic variation of the aqueous-phase NaOH concentration. Indeed, as shown in Figures 1 and 2, there is little apparent difference between the PHE extraction from simple sodium hydroxide solutions using 1 M 1-octanol and 15 wt% Exxal® 8 as modifier. The Na distribution coefficients are essentially identical at aqueous phase sodium hydroxide concentrations greater than 2 M.

Values of the sodium distribution ratio D_{Na} were used as input to the solvent extraction modeling program SXLSQI. Parameters and underlying assumptions used in the SXLSQI modeling are described elsewhere (Lumetta and Levitskaia, 2006). Based on the equilibrium reactions 1 through 4, the following organic-phase species were included in the computer model: Na^+, OH^-, NaOH, NaA, and NaA_2H. Because of the compositional and functional similarity of Isopar® L diluent modified with 1 M 1-octanol or Exxal® 8, the logarithmic values of the extraction constants of the reactions 1 and 2 were fixed and taken as corresponding values of -5.30 and -8.82 previously obtained for the 1-octanol/Isopar® L system (Lumetta and Levitskaia 2006). The logarithmic values of the extraction constants for the reactions 3 and 4 were refined to be -0.41±0.07 and 0.42±0.02 with the goodness of fit parameter of 1.65. Figure 2 presents a comparison of the sodium distribution values obtained experimentally and by the computer fit.

To determine the sodium extraction isotherm under conditions relevant to recycling sodium hydroxide from solutions generated from leaching high-level radioactive tank waste, a simulated waste leachate was prepared. There is no definitive recipe for preparing such simulated leachates. The composition used here was projected from the results of caustic leaching tests performed with radioactive tank waste sludges from the Hanford "S" tank farm (Lumetta et al. 1996, 1197, 2001). Leaching times of 8 h at 3 mL caustic solution per gram of sludge was assumed. The simulants were prepared such that the composition was as follows: 0.9 M sodium aluminate, 0.7 M sodium nitrate, 0.4 M sodium nitrite, 0.05 M sodium carbonate, 0.03 M sodium phosphate, 0.03 M sodium sulfate, and 1, 2, 3, 4, or 5 M sodium hydroxide. The sodium distribution coefficients obtained from simulated radioactive waste leachate are presented in Figure 2, where it is also compared with the values obtained from pure sodium hydroxide solutions.

PHE from the simulated waste leachate is enhanced compared to that from the pure sodium hydroxide solutions. For example, at an aqueous phase sodium hydroxide concentration of approximately 0.7 M, the distribution coefficient for PHE from simulated waste leachate (total sodium concentration of approximately 3 M) is a little over 50% greater than that for PHE from pure sodium hydroxide solution. The simulated waste leachate and pure sodium hydroxide solution isotherms converge at a sodium hydroxide concentration of 4.5 M, or a total sodium concentration of 6.75 M in the former solution. Clearly, the total aqueous phase sodium concentration affects the sodium hydroxide distribution coefficient. We found analyzing the data from the simulated waste leachate experiments with reference to the reactions 1 through 4 most useful in explaining these results.

Only reactions 3 and 4 are significant in the concentration range studied and their respective equations were solved to provide the following equation:

$$\left[Na^+\right]_{org} = \left[Na^+\right]_{aq} \gamma_{Na,aq} \left[OH^-\right]_{aq} \gamma_{OH,aq} \left(K_3 \left[HA\right]_{orf} + K_8 \left[HA\right]_{org}{}^2\right) \qquad (5)$$

In equation 5, $\gamma_{Na,\,aq}$ and $\gamma_{OH,\,aq}$ are the aqueous phase activity coefficients for Na^+ and OH^-, respectively and their product is referred to hereafter as the activity coefficient product, or $\dot{\gamma}$. The values for K_3 and K_4 are 0.389 and 2.63, respectively, as derived using SLXQI modeling. Therefore, results from the experiments with simulated waste leachate were considered in terms of activity coefficient product,

$$\dot{\gamma} = \frac{\left[Na^+\right]_{org}}{\left[Na^+\right]_{aq}\left[OH^-\right]_{aq}\left(K_3\left[HA\right]_{org} + K_8\left[HA\right]_{org}{}^2\right)} \quad (6)$$

Figure 3 compares the activity coefficient products derived from the results from the sodium hydroxide and simulated waste leachate experiments. That these data points are correlated by a single equation with good accuracy shows that the other metals and anions in the simulated waste leachate have insignificant impact on the sodium and hydroxide activity coefficients. The activity coefficient product is correlated by equation 7.

$$\dot{\gamma} = 0.126 + 0.868\exp\left(-1.33\left[Na\right]_{aq}\right) + 0.187\exp\left(-31{,}300\left[Na\right]_{aq}\right) \quad (7)$$

Note that the correlation was derived by forcing its result to approach unity as the total sodium concentration approaches zero. Equation 7 proved useful in deriving the conceptual counter-current flowsheet described below.

To demonstrate potential application of PHE, a preliminary process engineering evaluation was performed for PHE from a solution generated by caustic leaching of Hanford high-level tank waste solids. Material balances were formulated assuming PHE was performed in discrete extraction equipment, such as centrifugal contactors, and that the residence time was sufficient to achieve equilibrium in each stage. The equation empirically fitted to the pure sodium hydroxide isotherm (Figure 1) was used in preparing the material balances for the strip processes. Extraction and scrub process material balances were prepared using equations 6 and 7.

Extraction, scrub, and strip processes were considered in developing the flowsheet. PHE from the waste leachate occurs in the extraction process. In the scrub process, the organic phase product from the extraction process is contacted with 0.1 M sodium hydroxide to remove species of low extractability (e.g., other metal hydroxides). Such species have not yet been identified in testing performed to date. Tests to identify these species of low extractability would require either a more sophisticated simulated leachate or actual waste leachate. Once the extractability of these species has been determined then their impact on downstream uses of the separated sodium hydroxide can be assessed. A scrub process may not be needed if the impacts are insignificant. It is known that Cs^+ is extracted into the PHE solvent (Lumetta and Levitskaia 2006) but it is assumed that ^{137}Cs would previously be removed from the PHE feed to avoid contamination of the NaOH product with this radioisotope. In the strip process, sodium is back-extracted into water forming NaOH in the aqueous phase (the reverse of equation 3). The maximum allowable phase ratio in developing the material balances was ten. This phase ratio was considered practically reasonable in the absence of any hydrodynamic performance data. The material balances were developed limiting the minimum sodium hydroxide concentration in the aqueous raffinate to 0.1 M to prevent gibbsite precipitation.

Figure 4 illustrates one potential counter-current flowsheet and consists of extraction, scrub, and strip processes each consisting of 4 equilibrium stages. The organic-to-aqueous phase ratios are 4.6, 10 and 2.9 in the extraction, scrub, and strip sections, respectively. This flowsheet is predicted to recover 97% of the sodium hydroxide from the feed solution into a 1 M sodium hydroxide product. The flowsheet indicates the feasibility of the technology to PHE from chemically complex solutions, such as the radioactive waste leachate.

An experiment was performed to test the validity of the conceptual flowsheet described in Figure 4. The flowsheet experiment was essentially a co-current version of flowsheet performed

using batch equilibrium contacts (Figure 5). The entire volume of PHE solvent, based on the extraction phase ratio and aqueous phase volume, was split between the four extraction stages so that the aqueous phase was successively contacted with fresh PHE solvent. Likewise, the organic phase product from the first extraction stage was successively contacted with fresh scrub solution (0.1 M NaOH) and then strip solutions (water). The distribution behavior of sodium was determined radiometrically using ^{22}Na tracer. At equilibrium, aliquots of each phase were taken for gamma counting, and in selected cases aliquots of the aqueous phase was taken for hydroxide determination by titration with standard HCl.

The co-current flowsheet experiment demonstrated extraction of 82% of the sodium hydroxide in the simulated waste leachate in the four extraction contacts. Although the solution is super-saturated in Al after the final extraction contact, gibbsite precipitation was not observed during the course of the experiment. The kinetic stability of the solutions is adequate to run the PHE process, provided the free hydroxide concentration in the raffinate is maintained at a few tenths molar. Of the sodium hydroxide in the scrubbed organic phase, 75% was stripped in the four strip contacts. Therefore, the flowsheet experiment demonstrated the feasibility of applying PHE from chemically complex solutions using the solvent mixture consisting of 1.0 M 35-DTBP in Isopar® L modified with 15 wt% Exxal® 8.

Research Implications

Favorable isotherms for sodium extraction from pure sodium hydroxide solutions and simulated alkaline waste leachate show that a solvent mixture consisting of 1.0 M 35-DTBP in Isopar® L modified with 15 wt% Exxal® 8 is a potential candidate for recovery of sodium hydroxide from caustic solutions. A preliminary counter-current process flowsheet for sodium hydroxide extraction from radioactive waste leachate developed as part of this work demonstrates the feasibility of applying the technology. The flowsheet consists of four equilibrium stages in each of the extraction, scrub, and strip processes and recovers 97% of the feed sodium hydroxide. A co-current process flowsheet, based on the counter-current version, was experimentally demonstrated and its success further demonstrates PHE from complex caustic solutions is feasible. Implementation of a PHE process in conjunction with the caustic leaching process currently planned for pretreating Hanford tank sludges could result in a significant cost savings to the U.S. Department of Energy. This cost saving would be achieved through recycling of NaOH for use in sludge leaching, thus greatly reducing the amount of Na being added to the Hanford tank wastes.

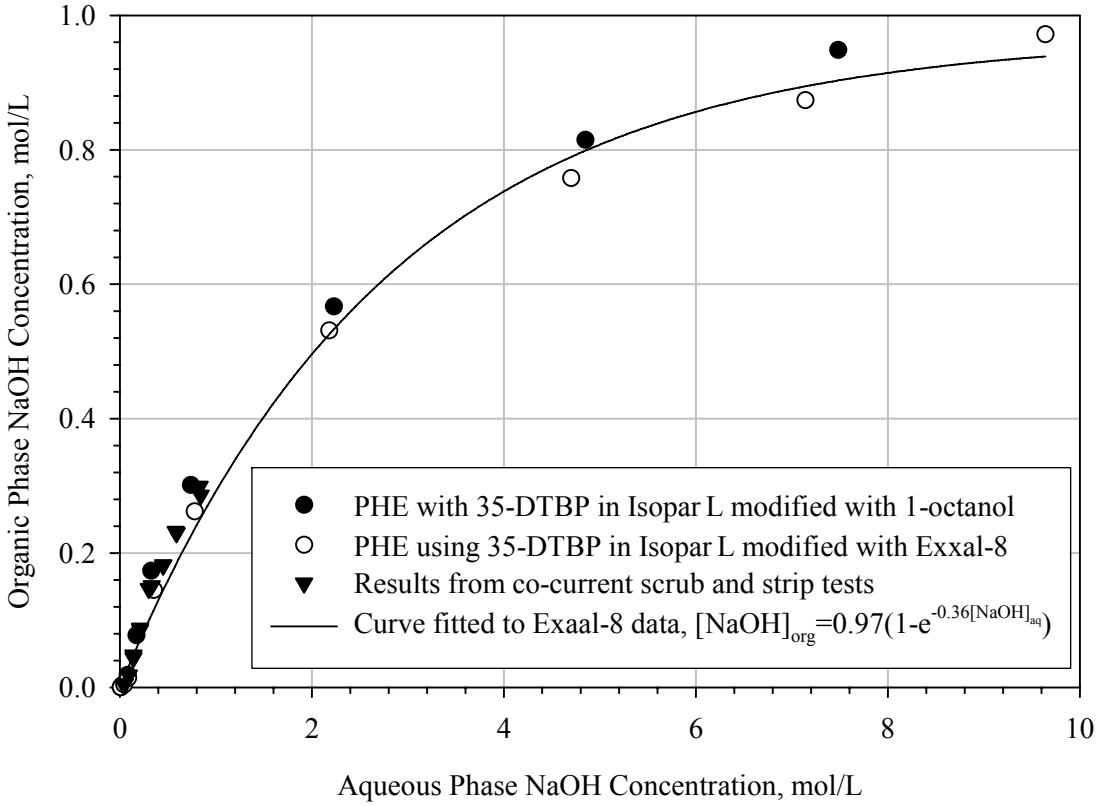

Figure 1. Comparison of equilibrium isotherms for PHE from pure sodium hydroxide solutions using 35-DTBP in Isopar® L modified with either 1-octanol or Exxal® 8

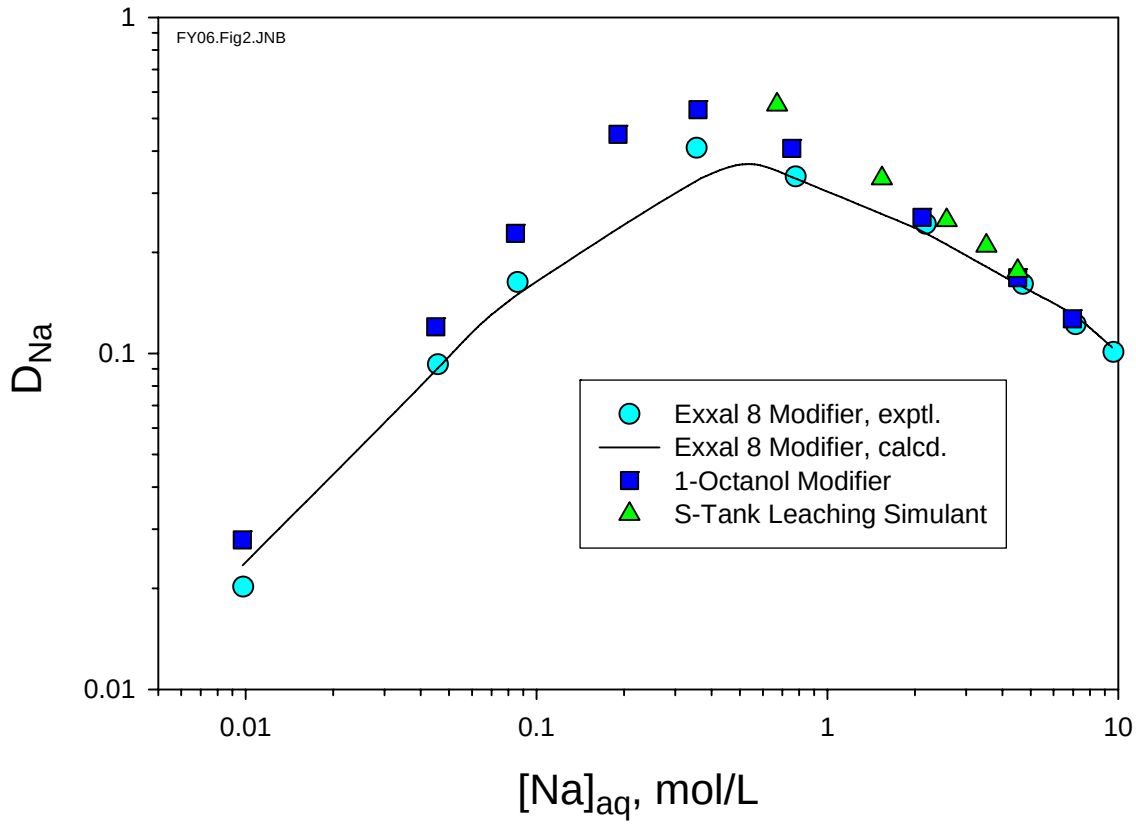

Figure 2. Comparison of sodium distribution coefficients for PHE from pure sodium hydroxide solutions and simulated radioactive waste leachate using 35-DTBP in Isopar® L modified with either 1-octanol or Exxal®-8; the calculated line was determined using the thermodynamic model described in the text.

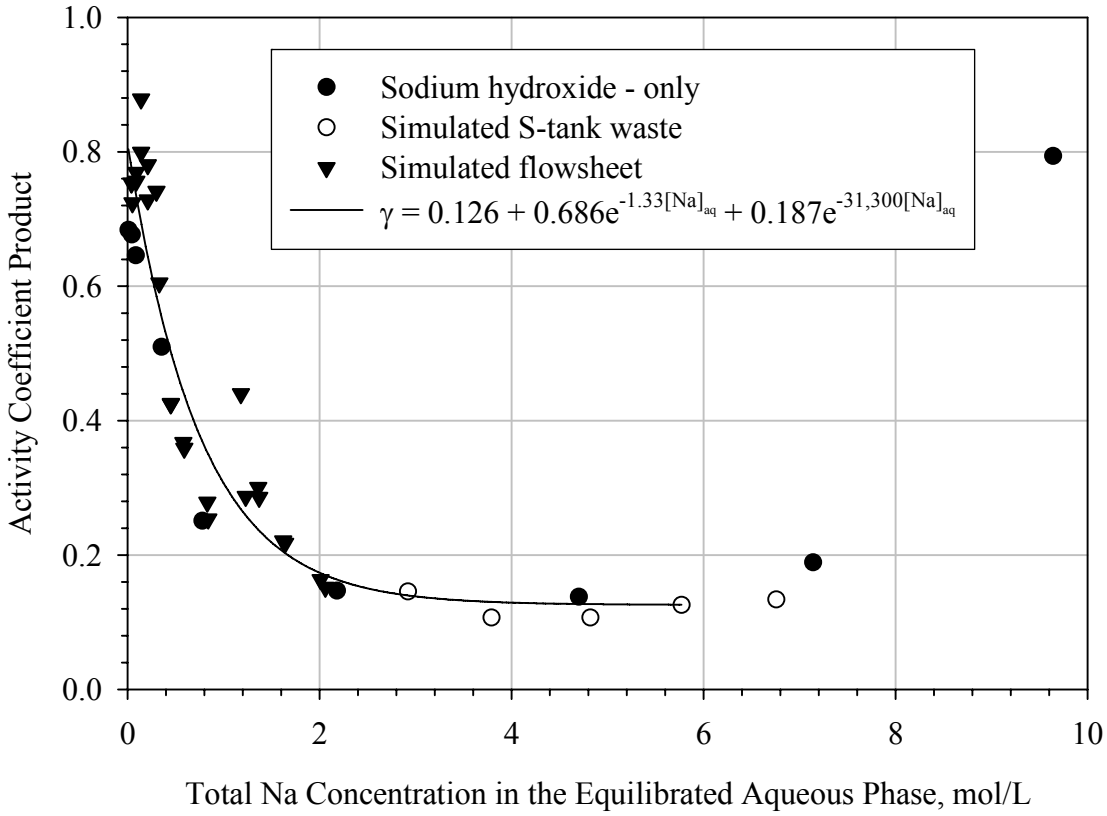

Figure 3. Comparison of activity coefficient products derived for PHE from pure sodium hydroxide solutions and simulated radioactive waste leachate using 3,5-DTBP in Isopar® L modified with Exxal® 8

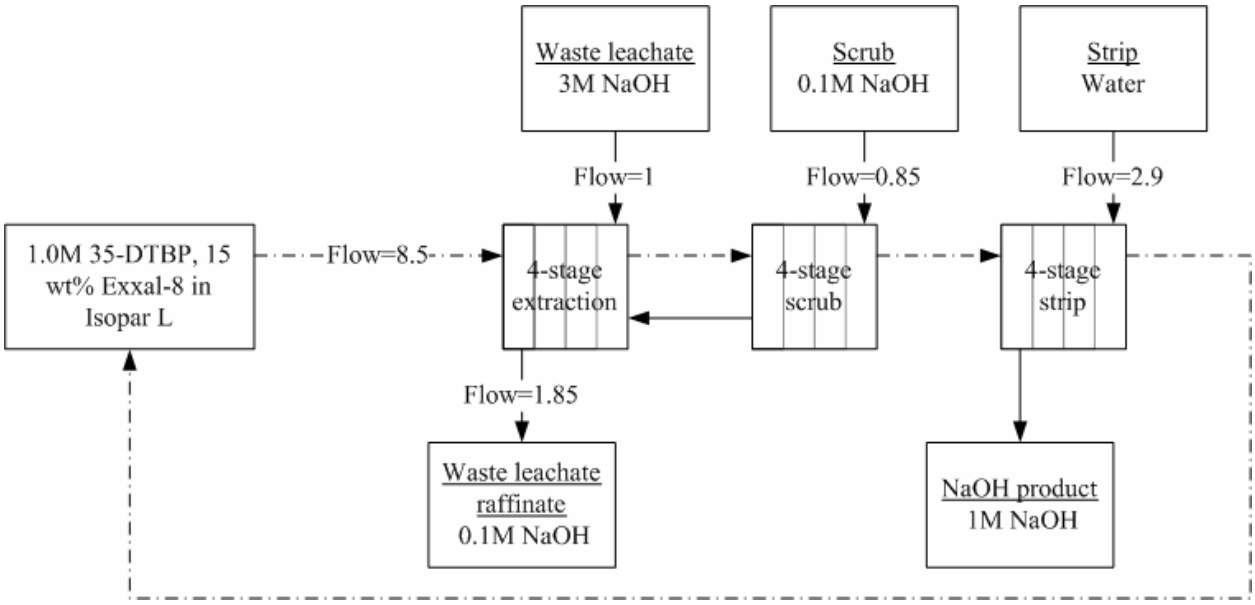

Figure 4. Conceptual process flowsheet for PHE from radioactive waste leachate

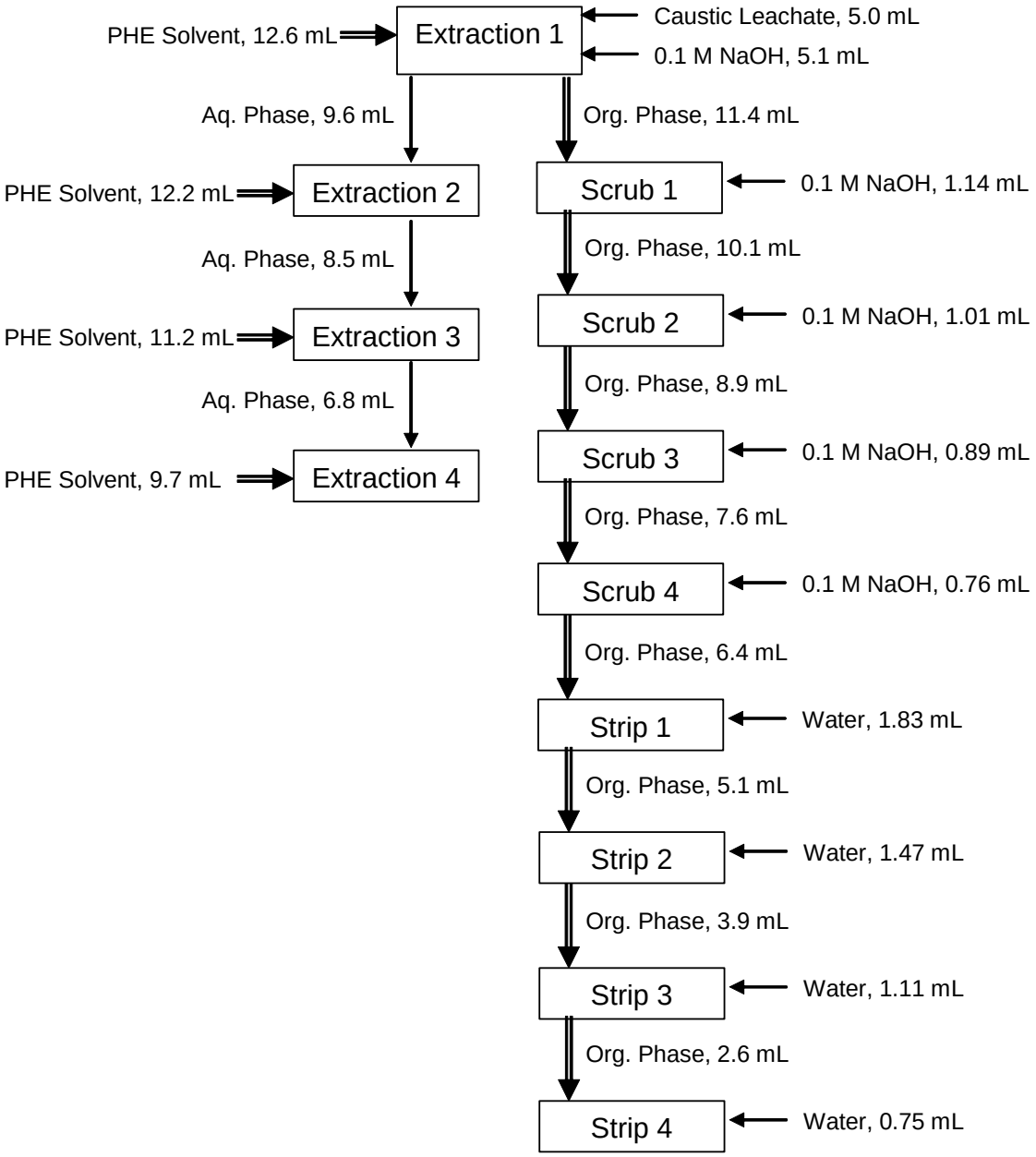

Figure 5. Schematic representation of batch equilibrium contacts performed to mimic the flowsheet depicted in Figure 4.

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